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7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Physics Department Queens College of CUNY Flushing, NY 11367			AFOSR-TR-96 0166	
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**THE SYNTHESIS OF MICROWAVE
MAGNETIC MATERIALS
AS ORIENTED SPUTTERED FILMS**

**F. J. Cadieu
Physics Department
Queens College of CUNY
Flushing, NY 11367**

**Date of Report
April 11, 1996**

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Air Force Office of Scientific Research
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Fig. 2. A x-ray diffractometer trace, $\text{Cu K}\alpha$ radiation, for a NiOFe_2O_3 film made onto a 930 °C C-plane sapphire substrate is shown. The oxygen pressure during deposition was 200 mTorr. Kikuchi backscattered electron diffraction has been used to show this is a single crystal film. J. Appl. Phys. **79**, 5425 (1996).

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Fig. 4. In plane hysteresis loops are shown along the hard and easy axes of a permalloy film deposited for device applications at Queens College. The inset figure shows the magnetoresistance measured along the in plane hard and easy axes. J. Appl. Phys. **73**, 5926 (1993).

Fig. 5. A SmCo based sputtered film has been used to bias the permalloy film. The geometry is shown in the lower part of the figure. The effective bias field can be determined by applying an in plane field at right angles to the bias field. When the magnetoresistive response has decreased to one half then the bias field and the applied field have the same value which allows the bias field value to be determined. The effective bias field in this case was 120 Oe. J. Appl. Phys. **73**, 5926 (1993).

Fig. 6. For SmCo based films directly deposited onto YIG substrates it has been possible to saturate the YIG film for SmCo to YIG thickness ratios of greater than 0.22. In this case as shown the SmCo based film was 118 μm thick. Boundary layers were required to obtain good film to substrate bonding for the thick films. J. Appl. Phys. **76**, 6059 (1994).

Papers Directly Resulting From
Air Force Office of Scientific Research Support As A Part of This Grant

1. F.J. Cadieu, "Permanent Magnet Thin Films" in Physics of Thin Films, Vol. 16, Academic Press, San Diego, May 15, 1992.
2. K. Chen, H. Hegde, and F.J. Cadieu, "Induced Anisotropy In Amorphous Sm-Co Sputtered Films", Applied Physics Letters, October 12, 1992.
3. K. Chen, H. Hegde, S.U. Jen, and F.J. Cadieu, "Different Types of Anisotropy in Amorphous SmCo Films", Paper CP-22, 37th Magnetism and Magnetic Materials Conference, Houston, December 1-4, 1992, J. Appl. Phys. **73**, 5923 (1993).
4. H. Hegde, S.U. Jen, K. Chen, and F.J. Cadieu, "Film Sm-Co Permanent Magnets for the Biasing of Thin Permalloy Strips", Paper CP-23, 37th Magnetism and Magnetic Materials Conference, Houston, December 1-4, 1992, J. Appl. Phys. **73**, 5926 (1993).
5. H. Hegde, P. Samarasekara, K. Chen, and F.J. Cadieu, "Non-Epitaxial Sputter Synthesis of Aligned Strontium Hexaferrite, $\text{SrO} \cdot 6(\text{Fe}_2\text{O}_3)$, Films", Paper FC-07 38th MMM, Minneapolis, November 15-18, 1993, J. Appl. Phys. **75**, 6640 (1994).
6. M. Levy, R. Scarmozzino, R.M. Osgood, F.J. Cadieu, and H. Hegde, "Permanent Magnet Film Magneto optic Waveguide Isolator", Paper DP-27 38th MMM, Minneapolis, November 15-18, 1993, J. Appl. Phys. **75**, 6286 (1994).
7. H. Hegde, P. Samarasekara, R. Rani, A. Navarathna, K. Tracy, and F. J. Cadieu, "Sputter Synthesis of TbCu_7 Type $\text{Sm}(\text{CoFeCuZr})$ Films With Controlled Easy Axis Orientation" Paper ER-22, 6th Joint MMM-INTERMAG, Albuquerque, June 20-23, 1994, J. Appl. Phys. **76**, 6760 (1994).
8. F. J. Cadieu, H. Hegde, E. Schloemann, and H. J. Van Hook, "In-Plane Magnetized YIG Substrates Self Biased by SmCo Based Sputtered Film Coatings", Paper AB-11, 6th Joint MMM-INTERMAG, Albuquerque, June 20-23, 1994, J. Appl. Phys. **76**, 6059 (1994).
9. F. J. Cadieu, Recent Advances in Pseudobinary Iron Based Permanent Magnets, International Materials Reviews **40**, 137 (1995).
10. F. J. Cadieu, High Energy Product Permanent Magnet Films, Invited Paper, 4th International Symposium on Magnetic Materials, Processes, and Devices, Electrochemical Society Meeting, Chicago, October 8-13, 1995, Proceedings Electrochemical Society (to be published).
11. H. Hegde, X. R. Qian, Jong-Guk Ahn, and F. J. Cadieu, High Temperature Magnetic Properties of TbCu_7 -type SmCo Based Films, Paper FF-09, 40th Magnetism and Magnetic Materials Conference, Philadelphia, Nov. 6-9, 1995, J. Appl. Phys. **79**, 5961 (1996).

12. P. Samarasekara, R. Rani, F. J. Cadieu, and S. A. Shaheen, Variable Texture NiOFe_2O_3 Ferrite Films Prepared By Pulsed Laser Deposition, Paper DP-02, 40th Magnetism and Magnetic Materials Conference, Philadelphia, Nov. 6-9, 1995, J. Appl. Phys. **79**, 5425 (1996).
13. M. Levy, R. M. Osgood, Jr., H. Hegde, F. J. Cadieu, R. Wolfe, and V. J. Fratello, Integrated Optical Isolators With Sputter-Deposited Thin-Film Magnets, Photonics Technology Letters (to be published, July 1996).

Of these papers the following were invited longer papers and reviews.

1. F.J. Cadieu, "Permanent Magnet Thin Films" in Physics of Thin Films, Vol. 16, Academic Press, San Diego, May 15, 1992.
9. F. J. Cadieu, Recent Advances in Pseudobinary Iron Based Permanent Magnets, International Materials Reviews **40**, 137 (1995).
10. F. J. Cadieu, High Energy Product Permanent Magnet Films, Invited Paper, 4th International Symposium on Magnetic Materials, Processes, and Devices, Electrochemical Society Meeting, Chicago, October 8-13, 1995, Proceedings Electrochemical Society (April 1996, to be published).

III. Scientific Personnel

1. Professor Fred J. Cadieu, Physics Department, Queens College of CUNY.
2. Dr. Hari Hegde, Physics Department, Queens College of CUNY.
3. Dr. Raj Rani, Physics Department, Queens College of CUNY.
4. Professor X. R. Qian, Visiting Scholar, Professor Qian from the Shanghai Iron and Steel Research Institute was in part supported by this grant during 1994. Dlr. Qian obtained permanent U. S. residency in July 1995 while working as a part of this grant.
5. Professor Shahid A. Shaheen, Physics Department, Florida State University. Professor Shaheen contributed to the progress of some of this project while I was on sabbatical at the Center for Materials Research and Technology, Florida State University, from August 1995 to July 1996.
6. Mr. Kailai Chen, Ph.D. Candidate Thesis Student, Graduate Center of CUNY, and Physics Department, Queens College of CUNY.

7. Mr. Anil Navarathna, Ph.D. Candidate Thesis Student, Graduate Center of CUNY, and Physics Department, Queens College of CUNY.
8. Mr. Pubudu Samarasekara, Ph.D. Candidate Thesis Student, Graduate Center of CUNY, and Physics Department, Queens College of CUNY. Mr. Samarasekara was supported in part from this grant while working on this project at the Center for Materials Research and Technology, Florida State University, Tallahassee, FL 32306.

IV. Degrees Awarded

1. Mr. Kailai Chen received the Ph.D. degree from the City University of New York at the May 1993 commencement.
2. Ms. Raj Rani received the Ph.D. degree from the City University of New York at the January 1995 commencement.
3. Mr. Anil Navarathna received the Ph.D. degree from the City University of New York at the May 1995 commencement.
4. Mr. Pubudu Samarasekara received the Ph.D. degree from the City University of New York at the January 1996 commencement.

The Synthesis of Microwave Magnetic Materials As Oriented Sputtered Films

F. J. Cadieu

Physics Department

Queens College of CUNY

Flushing, NY 11367

Abstract

Significant results were made in the synthesis of ferrite films by both sputtering methods and by pulsed laser ablation. An initial result of this program resulted in the nonepitaxial synthesis of strontium ferrite films with the crystallite c-axes aligned onto the substrate plane. For substrate temperatures near the comparatively low temperature of 500 °C, the films consisted of random in-plane c-axes grains. In later studies, nickel ferrite films with various textures were deposited onto different substrates by pulsed laser ablation. Nickel ferrite films deposited onto c-plane sapphire were single crystal films with a (111) orientation. Currently a pulsed laser deposition system is being purchased for installation at Queens College through a recently funded NSF ARI grant. In other parallel studies, SmCo based films exhibiting the TbCu₇-type structure were synthesized and studied for a number of device type applications. SmCo based films with energy products of approximately 16 MGOe were synthesized for the biasing of permalloy films and YIG substrates. In other studies, SmCo based permanent magnet films were used in the fabrication of a film based Bi-YIG magneto-optical waveguide isolator. Isolation ratios of 28-30 dB were obtained for light wavelengths from 1490-1555 nm. It was also shown that the SmCo based films with the TbCu₇-type structure exhibited very good high temperature magnetic properties and could be reversibly cycled to at least 460 °C without deteriorating the room temperature magnetic properties. The anisotropy field for magnetizing the films in-plane versus perpendicular to the plane was ≈ 18 kOe at 460 °C.

Significant Research Advances

I. The Deposition of Ferrite Films By Sputtering And By Pulsed Laser Ablation

- a. "Non-Epitaxial Sputter Synthesis of Aligned Strontium Hexaferrite, $\text{SrO} \cdot 6(\text{Fe}_2\text{O}_3)$, Films", J. Appl. Phys. **75**, 6640 (1994).

Crystalline films of strontium hexaferrite were synthesized non-epitaxially by RF diode sputtering in Ar on to polycrystalline Al_2O_3 substrates for substrate temperatures from 500 to 650 °C. To counter severe Sr depletion during sputtering, targets highly enriched in Sr were utilized. After deposition large increases in coercivity up to 3.8 kOe, but with no apparent changes in x-ray patterns, resulted from annealing the films in 500 mTorr O_2 . The films produced in these studies exhibited crystallites with well defined c-axes orientations. For deposition temperatures near 500 °C, the films consisted of random in-plane c-axes grains. For deposition temperatures from 600 to 650 °C, the films consisted of grains with random c-axes orientations in three dimensions. The maximum process temperatures used in this study were lower than those usually used for the crystallization of ferrite films.

b. Pulsed Mode Sputtering For Higher Rate Deposition of Ferrite Films

Two methods have been used to synthesize ferrite films of principally Ni and Li ferrite. The first method has been sputtering which suffers from unusually low rates for making ferrite films. The other method used has been pulsed laser deposition, PLD.

To circumvent the low deposition problem for sputtered films, a pulsed RF sputtering method has been developed that allows insulating ferrite materials to be deposited at deposition rates comparable to those normally only available for metallic films. In this method the RF applied power is automatically sequentially stepped between two power levels. Normally sputtering deposition rates for ferrites are only ≈ 0.1 to 0.2 \AA/s .

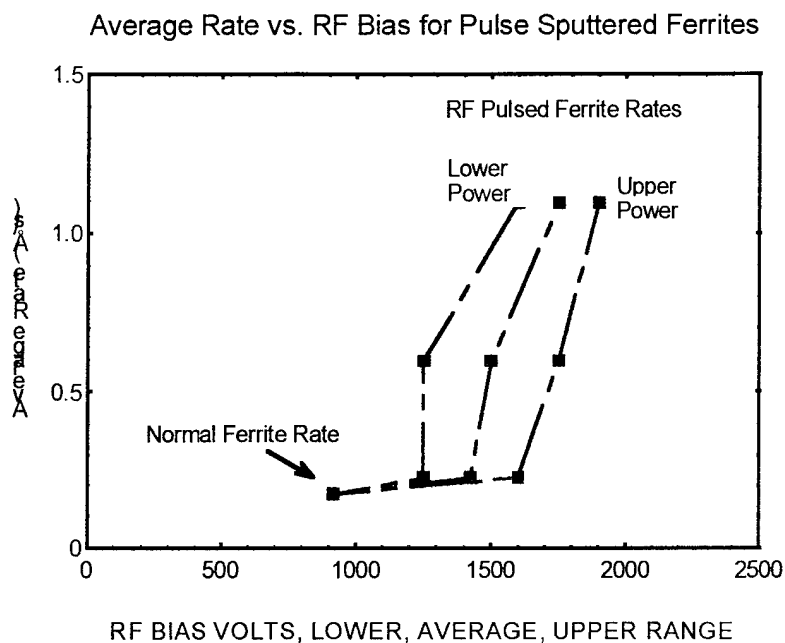


Fig. 1. A method was developed using pulsed sputtering powers switched between a high and a low level which acted to greatly increase the rate of ferrite film deposition by sputtering. Normally the sputtering rates available for sputtered films are well below those normally attained for metallic films.

**c. VARIABLE TEXTURE NiOFe_2O_3 FERRITE FILMS
PREPARED BY PULSED LASER DEPOSITION**

J. Appl. Phys. **79**, 5425 (1996).

Abstract

Pulsed laser deposition using bulk NiOFe_2O_3 targets onto heated substrates has been used to synthesize single phase Ni spinel ferrite films onto various substrates. Flowing oxygen has been used during the deposition to maintain the oxygen stoichiometry. Ni ferrite films have been deposited onto C, R, and A-plane sapphire, polycrystalline Al_2O_3 , and fused silica. Single crystal (111) oriented NiOFe_2O_3 films have been made onto C-plane sapphire for substrate temperatures of greater than 900 °C. Polycrystalline highly (400) textured Ni ferrite films have been made onto R-plane sapphire with $I(400)/I(311) = 6.15$. Films made onto A-plane sapphire, polycrystalline alumina, and fused silica showed only moderate texturing. The coercive force of the (111) oriented NiOFe_2O_3 was 120 Oe perpendicular to the film plane and 95 Oe in plane.

The texture modes grown by using the different substrates are summarized in Table 1. The most important results of this paper are that single crystal NiOFe_2O_3 films can be grown by pulsed laser deposition with (111) orientation onto C-plane sapphire, and that highly (400) textured polycrystalline NiOFe_2O_3 films can be grown onto R-plane sapphire substrates. Single crystal (111) oriented NiOFe_2O_3 films exhibit three fold symmetry in the substrate plane and d_{111} exceeds the a lattice parameter of Al_2O_3 by only 1.18%. These conditions allow the growth of single crystal (111) oriented NiOFe_2O_3 films onto C-plane sapphire. Relatively high substrate temperatures of ≥ 900 °C have been required to grow completely oriented and or textured films. The single crystal (111) oriented films are of special interest since the easy direction of magnetization for NiOFe_2O_3 is along that direction.

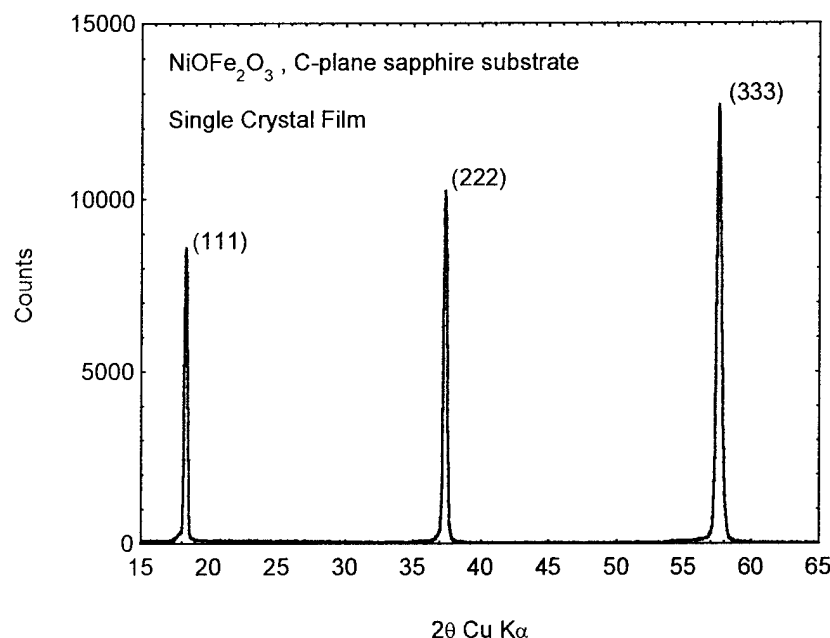


Fig. 2. A x-ray diffractometer trace, Cu $K\alpha$ radiation, for a NiOFe_2O_3 film made onto a 930 °C C-plane sapphire substrate is shown. The oxygen pressure during deposition was 200 mTorr. Kikuchi backscattered electron diffraction has been used to show this is a single crystal film. J. Appl. Phys. **79**, 5425 (1996).

Table 1. The dominant NiOFe_2O_3 x-ray texture for different substrates is shown for pulsed laser deposition with 200 mTorr O_2 and high substrate temperatures. J. Appl. Phys. **79**, 5425 (1996).

Substrate	Ni-Ferrite Dominant Texture	I(400)/I(311) Intensity Ratio
C-Plane Sapphire (006)	(111), (222), (333) only single crystal films	neither detected
A-Plane Sapphire (110)	(311)	1.20
R-Plane Sapphire(012)	(400)	6.15
Fused Silica	Random, (311)	0.400
Polycrystalline Al_2O_3	(311) as deposited, after flash heating in air,	$I(220)/I(311) = 0.544^*$ $I(220)/I(311) = 0.433^*$

* For the polycrystalline Al_2O_3 substrates the intensity ratio for the I(220) reflection is used because of the strong Al_2O_3 (113) peak which is nearly coincident with the NiOFe_2O_3 (400) peak.

II. The Sputter Synthesis of SmCo Based Films For Device Type Applications.

a. Sputter Synthesis of TbCu₇ Type Sm(CoFeCuZr) Films With Controlled Easy Axis Orientation, J. Appl. Phys. **76**, 6760 (1994).

This paper dealt with the synthesis of highly textured films of SmCo based films with the crystallite c-axes aligned onto the film plane. Such films exhibit an extreme degree of in-plane anisotropy. Typically such films optimally prepared have remanent flux densities of 8-8.5 kG, intrinsic coercivities of 6-8 kOe, and room temperature in-plane static energy products of 16-19 MGOe. The Curie temperature of this system is approximately 700 °C so that such films are useful to relatively high temperatures. One of the results of this paper is the advantages of using Ar-Xe sputtering gas mixtures to maximize the degree of in-plane anisotropy and the remanent magnetization.

Such films have been used to fabricate several types of small scale device geometries. Films with thicknesses up to 120 μm have been deposited onto polycrystalline aluminum oxide, Si, and GaAs substrates. Boundary layers have been necessary to obtain film to substrate adhesion for films thicker than about 12 μm.

b. H. Hegde, X. R. Qian, Jong-Guk Ahn, and F. J. Cadieu, High Temperature Magnetic Properties of TbCu₇-type SmCo Based Films, Paper FF-09, 40th Magnetism and Magnetic Materials Conference, Philadelphia, Nov. 6-9, 1995, J. Appl. Phys. **79**, 5961 (1996).

Work on TbCu₇-type SmCo based films has been continued under U. S. Army Research Office support. The high temperature magnetic properties of this system have been prepared for presentation at the 40th MMM Conference to be held in Philadelphia, November 6-9, 1995. Normally bulk samples in this composition range form the two phase cellular structure whose magnetic properties are very sensitive to heat treatments. Single phase TbCu₇-type films of composition Sm₁₃Co₅₈Fe₂₀Cu₇Zr₂ were sputter synthesized such that the crystallite c-axes were oriented in the film plane. Optimal magnetic properties are obtained from the as sputtered films. The magnetic properties of these single phase films are insensitive to subsequent thermal heat treatments. The net anisotropy field of the films remained larger than the maximum applied field of 18 kOe even at the highest measurement temperature of 460 °C. Thus all measurements were from minor hysteresis loops. The in plane coercivity showed a monotonic decrease from 6.0 kOe at room temperature to 1.3 kOe at 460 °C. The in plane remanent flux density decreased from 8.5 kG at 50 °C to 5.8 kG at 460 °C.

The coercivity, remanent induction, and flux density at an applied field of 18 kOe for a SmCo based TbCu₇ type film as a function of temperature are shown in the figure below.

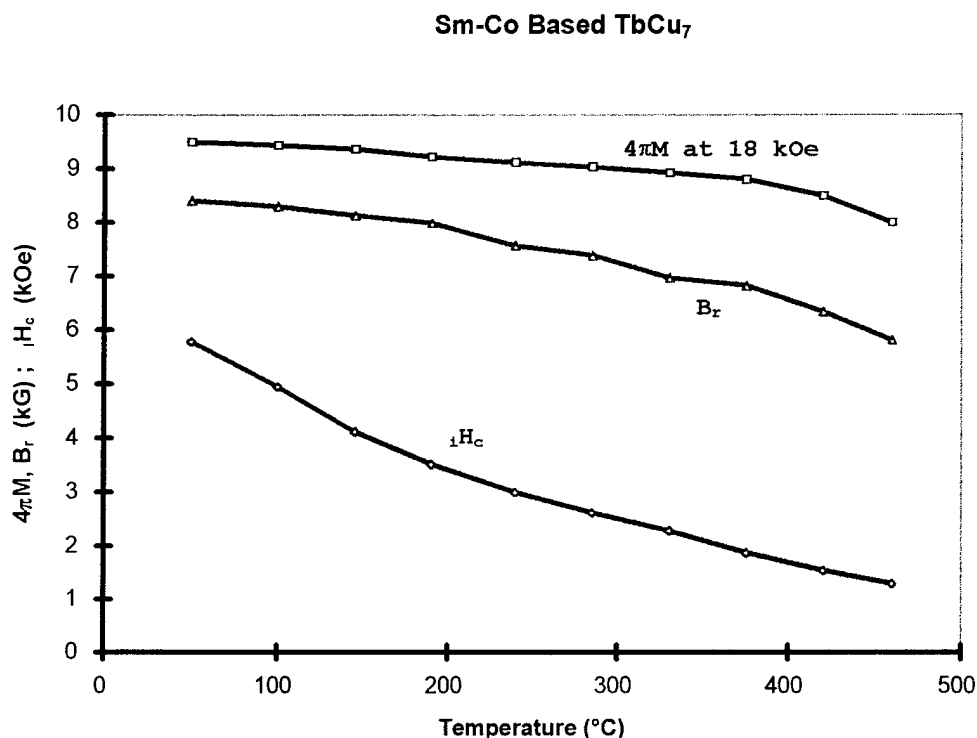


Fig. 3. Temperature dependence of in plane coercivity H_c , in plane B_r , and in plane $4\pi M$ at 18 kOe for a c-axes in plane textured SmCo based TbCu₇-type film are shown as a function of temperature up to 500 °C. J. Appl. Phys. **79**, 5961 (1996).

c. Paper CP-23 MMM'92, J. Appl. Phys. **73**, 5926 (1993).

FILM Sm-Co PERMANENT MAGNETS FOR THE BIASING OF THIN PERMALLOY STRIPS

H. Hegde, S.U. Jen*, K. Chen, and F.J. Cadieu,

Department of Physics, Queens College of CUNY, Flushing, NY 11367.

Abstract

Highly textured Sm-Co based permanent magnet films which exhibit in-plane anisotropy, with thicknesses ranging from 500 Å to several microns, have been synthesized on polycrystalline Al₂O₃ substrates. The textured Sm-Co based films of the disordered TbCu₇ type structure were directly crystallized by sputter deposition onto heated substrates. Remanent inductions, B_r , were ~8 kG, and the intrinsic coercivities, H_c , ranged from 4 to 10 kOe. The crystallite grain size as a function of film thickness

was determined. The use of these films in small scale geometry devices was tested in a sandwich configuration of strips of areal size $7 \times 1 \text{ mm}^2$. The Sm-Co films were used to bias $\text{Ni}_{81.5}\text{Fe}_{18.5}$ permalloy films. The sandwich permanent magnet was magnetized across its width. The permalloy films were sputter synthesized under conditions very similar to those of the permanent magnet films. Substrate biasing was not used for either the permanent magnet or the permalloy strips. The permalloy films exhibited a well-defined easy axis along the length of the strip. Unbiased, the permalloy showed a maximum magnetoresistance MR_{max} of 3.0 %. On biasing with a $25 \text{ }\mu\text{m}$ thick permanent magnet film with a spacer thickness of $25 \text{ }\mu\text{m}$, the longitudinal biasing field value at $\text{MR}/\text{MR}_{\text{max}} = 0.5$ was more than a 100 Oe, indicating an average biasing field of the same value.

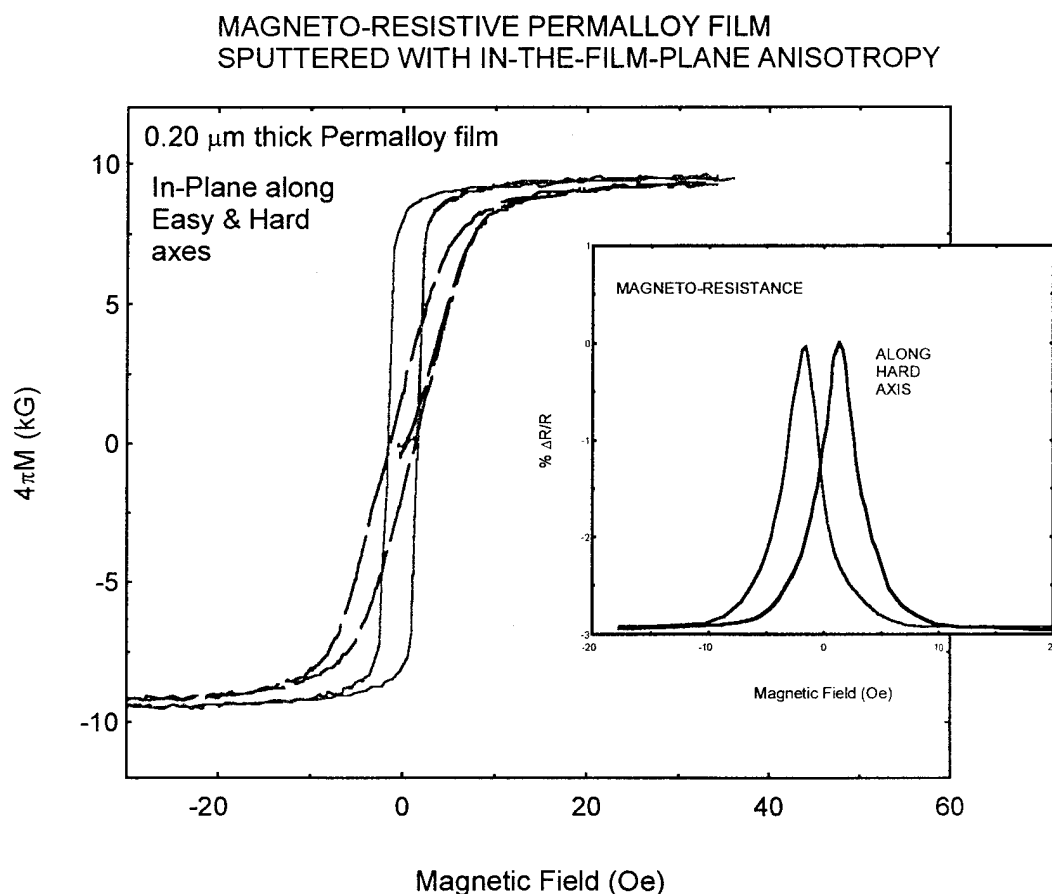


Fig. 4. In plane hysteresis loops are shown along the hard and easy axes of a permalloy film deposited for device applications at Queens College. The inset figure shows the magnetoresistance measured along the in plane hard and easy axes. J. Appl. Phys. **73**, 5926 (1993).

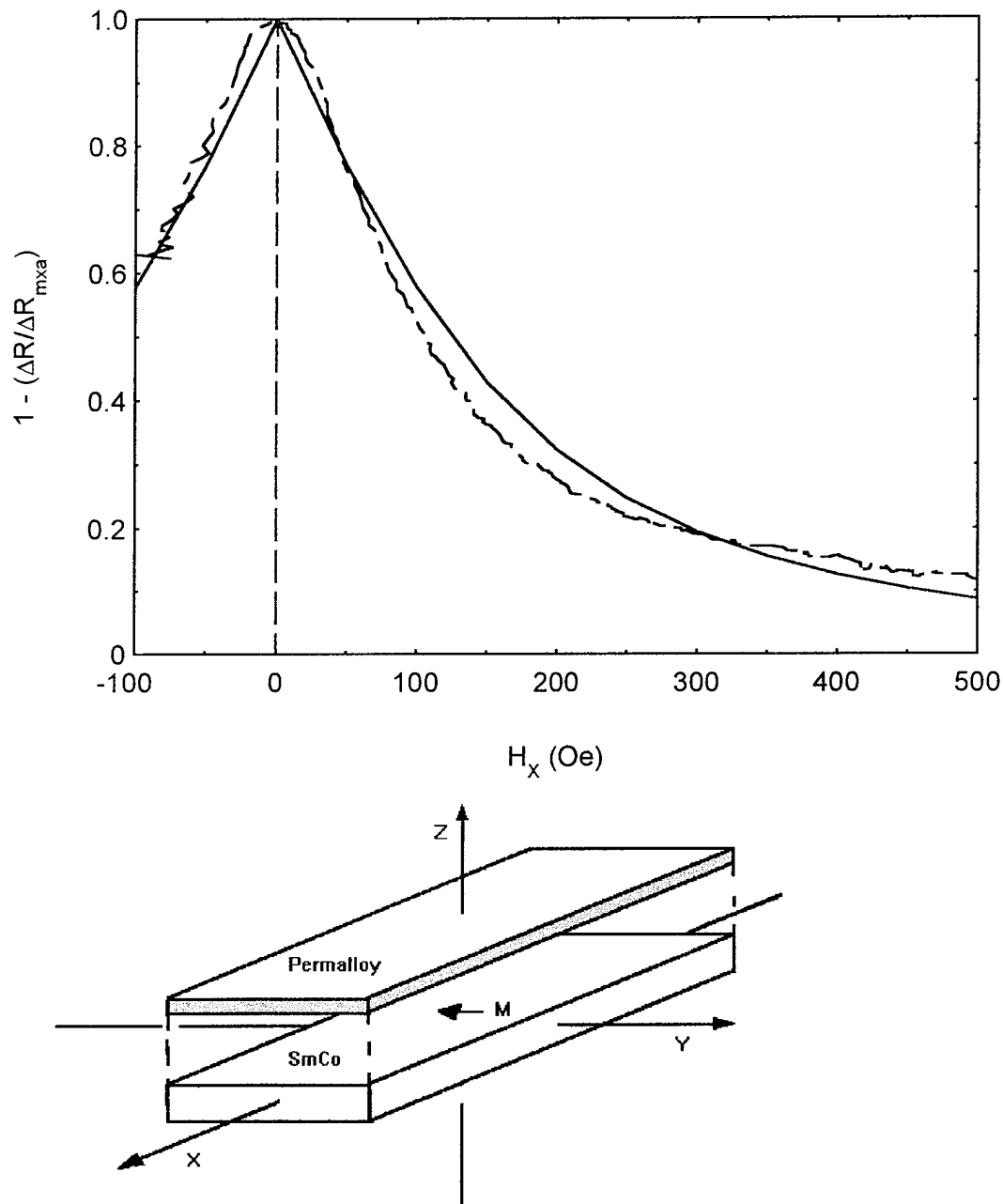


Fig. 5. A SmCo based sputtered film has been used to bias the permalloy film. The geometry is shown in the lower part of the figure. The effective bias field can be determined by applying an in plane field at right angles to the bias field. When the magnetoresistive response has decreased to one half then the bias field and the applied field have the same value which allows the bias field value to be determined. The effective bias field in this case was 120 Oe. *J. Appl. Phys.* **73**, 5926 (1993).

d. In-Plane Magnetized YIG Substrates Self Biased By SmCo Based Sputtered Film Coatings, J. Appl. Phys. 76, 6059 (1994).

SmCo based films exhibiting in-plane anisotropy were deposited onto YIG substrates. A boundary layer was used so that highly adherent in-plane anisotropy SmCo based films with thicknesses from 80 to 120 μm could be deposited onto YIG and other substrates. It was observed that for SmCo to YIG thickness ratios of greater than 0.22 that the looping field from the SmCo film layer was sufficient to saturate the YIG magnetization in the reverse direction. Such a geometry can also be used to reverse saturate patterned regions of a YIG substrate.

The deposition of strongly aligned in-plane anisotropy permanent magnet films onto YIG substrates was mainly supported by a subcontract as a part of the Ferrite Development Consortium.

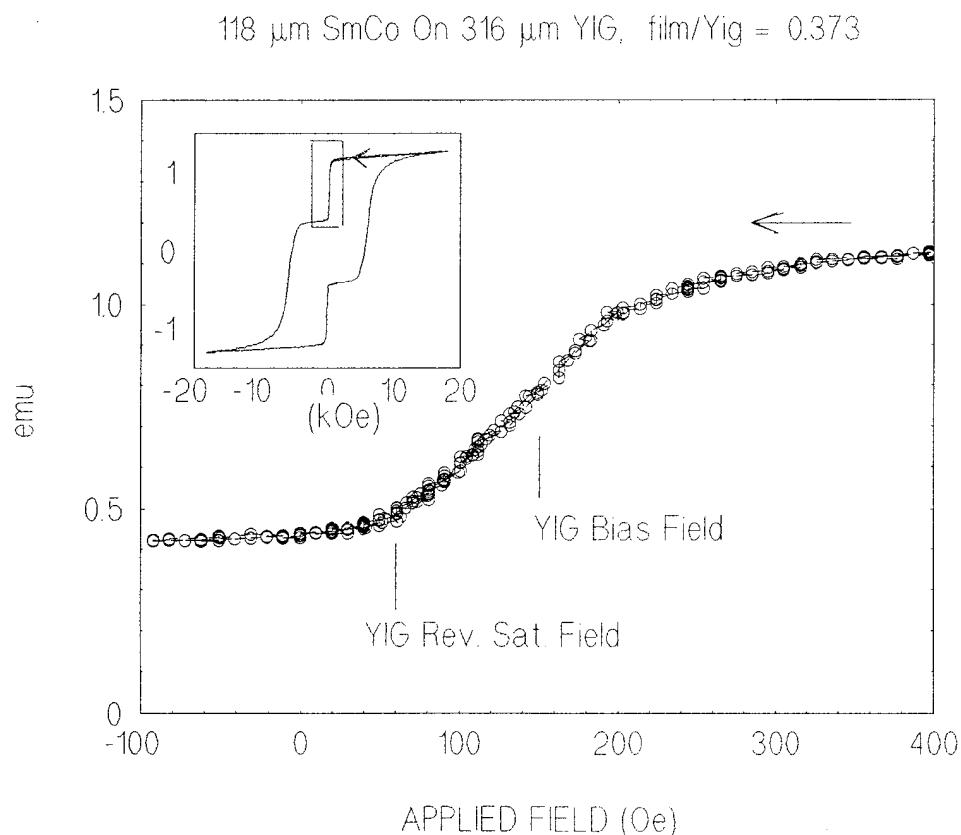


Fig. 6. For SmCo based films directly deposited onto YIG substrates it has been possible to saturate the YIG film for SmCo to YIG thickness ratios of greater than 0.22. In this case as shown the SmCo based film was 118 μm thick. Boundary layers were required to obtain good film to substrate bonding for the thick films. J. Appl. Phys. 76, 6059 (1994).

e. Induced Anisotropy in Amorphous SmCo Based Films

The following two papers were published that showed it was possible to synthesize films that exhibited a true uniaxial anisotropy within the plane of a film. In this case the in-plane loop measured along the direction of a magnetic field that had been applied during the sputter deposition was rectangular, while the in-plane loop measured orthogonal to that direction was completely closed and exhibited no coercivity.

2. K. Chen, H. Hegde, and F.J. Cadieu, "Induced Anisotropy In Amorphous Sm-Co Sputtered Films", Applied Physics Letters, October 12, 1992.
4. K. Chen, H. Hegde, S.U. Jen, and F.J. Cadieu, "Different Types of Anisotropy in Amorphous SmCo Films", Paper CP-22, 37th Magnetism and Magnetic Materials Conference, Houston, December 1-4, 1992, J. Appl. Phys. **73**, 5923 (1993).

f. PERMANENT MAGNET FILM MAGNETOOPTIC WAVEGUIDE ISOLATOR

In Conjunction With: Columbia University Microelectronics Sciences Laboratory,
AT&T Bell Laboratories, and the Naval Research Laboratory

As a part of this research a film based high performance magnetooptic waveguide isolator has been constructed and tested. In a preliminary device configuration consisting of a waveguide etched into a Bi-YIG film, a SmCo based permanent magnet film deposited onto a separate substrate was used to saturate the magnetization of the Bi-YIG waveguide. The TbCu₇ SmCo based film magnet was 22 μm thick in this case. A 45° rotation for a 1550 nm light wavelength was provided by a Bi-YIG light-pipe length of 3.55 mm with measured isolation ratios of 25 dB for light wavelengths from 1490 to 1555 nm. The publication citation for this paper was -- M. Levy, R. Scarmozzino, R.M. Osgood, F.J. Cadieu, and H. Hegde, "Permanent Magnet Film Magnetooptic Waveguide Isolator", Paper DP-27 38th MMM, Minneapolis, November 15-18, 1993, J. Appl. Phys. **75**, 6286 (1994).

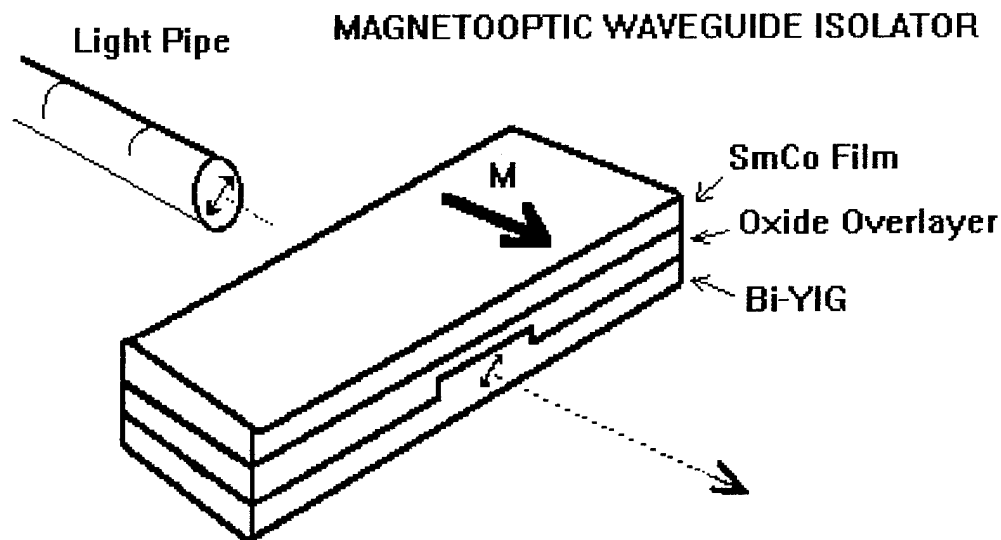


Fig. 6. In a subsequent paper a $7.5\text{ }\mu\text{m}$ thick SmCo based film was deposited directly onto the Bi-YIG waveguide structure. A thin boundary layer of Al was deposited to eliminate thermal stresses. Negligible insertion losses were measured for the actual device structure. Photonics Technology Letters (to be published, July 1996).

In the following paper a $7.5\text{ }\mu\text{m}$ thick SmCo film with suitable boundary layers was deposited directly onto the Bi-YIG waveguide. This was accomplished without any additional insertion losses. The isolation ratio was 28-30 dB over the light wavelength of 1490 to 1555 nm. This paper is scheduled to be published shortly in Photonics Technology Letters -- M. Levy, R. M. Osgood, Jr., H. Hegde, F. J. Cadieu, R. Wolfe, and V. J. Fratello, "Integrated Optical Isolators With Sputter-Deposited Thin-Film Magnets", Photonics Technology Letters (to be published, July 1996).